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Dept. of the Army Project No. 1C 522301 A 065

RMD Project No. 5802

SECOND QUARTERLY TECHNICAL REPORT

Covering the Period
15 September - 14 December 1964

Title: FIELD CALORIMETRY/CHEMICAL STUIDES

Sponsored by:

ADVANCED RESEARCH PROJECTS AGENCY
CHEMISTRY OFFICE
ARPA Order No. 535, Task e
Program Code No. 4860

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Prepared by:

EARL C. KLAUBERT
6 January 1965

Thiokol
CHEMICAL CORPORATION
REACTION MOTORS DIVISION
DENVILLE, NEW JERSEY

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Second Quarterly Report, 15 September - 14 December 1964
Field Calorimetry/Chemical Studies
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Phone: Area Code 201 - 627-7000 Ext. 6022

Major contributions to the work reported herein were made by Messrs, R. H. Tromans, J. Kahrs, and H. Francis.

Submitted by: Earl C. Klaubert
Earl C. Klaubert
Project Engineer

Approved by: Albert D. Corbett, Jr.
Albert D. Corbett, Jr.
Section Supervisor

Steven J. Tunkel
Steven J. Tunkel
Manager
Research Engineering Dept.

David J. Mann
David J. Mann
Director of Research

ERRATA SHEET FOR PRECEDING REPORT

First Quarterly Technical Report, 15 June - 14 September 1964
Field Calorimetry/Chemical Studies
ARPA Order No. 535, Task e; Program Code No. 4860
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Omitted from approval page (first page inside cover, no number):

"Major contributions to the work described herein were made by Messrs. R. H. Tromans, H. Francis, and K. D. Hill."

Page 22: last line omitted; should read

"and $\Delta h_r = \int_0^{\tau} [Q_r(t)] d\tau$ where τ = time."

Page 24: paragraph 2, line 7: symbol W omitted at right end of line; should read

". . . is increasing while W . . . "

Page 28, line 9: symbol W omitted; should read

". . . of W and of geometry ."

ABSTRACT

The purpose of this program is to develop a methodology by which to define quantitatively the heat flux from fires of flame fuels burning under field conditions. Quantitative work during this period involved indoor flames of hexane and napalm test solvent in diameters of 11, 18, and 22½ inches. Energy emitted in the visible region was compared with total radiant emission. Preliminary heat balances were conducted on the larger two sizes of flames each with the two fuels; very good agreement was achieved. Heat balances also were made on fires subjected to artificial low-velocity "winds", and spectra and burning rate were measured for gelled test solvent. The first lots of gelled fuel were prepared, and preliminary tests of the proposed explosive dissemination technique were conducted. Weather conditions prevented quantitative measurements on large outdoor fires. A system of foreoptics to provide either thermopile or spectrometer with a narrow field of view has been designed and is being fabricated.

Difficulties resulting from fluctuations in flames, and the need for evaluating fuels under steady-state conditions by measuring heat release rates are discussed.

Work for the ensueing quarter is outlined.

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1. INTRODUCTION

The purpose of this program is to develop an instrumentation system which will define qualitatively and quantitatively the modes by which the energy of combustion of a fuel is released when burned in large fires in open field environment. Consideration is to be given to the unreleased chemical energy contained in the partially-oxidized fuel components (soot, CO, and hydrocarbon compounds) discharged from the fire, and to convective energy contained in the no longer radiating hot gas column above the fire, as well as to radiated energy and the spectral distribution thereof.

Development of the instrumentation technique will be accomplished principally with a standardized technique for uniform dissemination of a standardized hydrocarbon fuel (napalm test solvent). Subsequently, the napalm flamethrower system (with standardized fuel and with conventional gelled gasoline) will be evaluated. Finally, provision will be made for testing of an optimized fuel toward the end of the program.

2. Experimental Work

The experimental work during this quarter was restricted mainly to indoor fires in order to gather additional background data, to complete fabrication of certain facilities and instrumentation, and because of weather conditions. The work accomplished is discussed in the following sections.

2.1 Visible Light vs. Total Radiant Emission

The validity of determining radiation source size and shape by photographic means involves, in part, the question of the percentage of total radiated energy which will be observed by this means. A convenient measurement could be made of the ratio of visible light (such as would be recorded by color film) to total radiation by means of a conventional photographic exposure meter. These meters respond principally to that portion of the spectrum to which the film is sensitive, and are relatively insensitive even to near-infrared radiation. The estimation of visible energy from a one-point measurement was based upon an admittedly crude dimensional model of the radiating flame, but was intended to be conservative (i.e., to yield a radiated visible power figure which would, if anything, be in error on the high side). From an 18-inch diameter fire of liquid napalm test solvent (NT3), the calculated visible power emission was about 180 watts; while Table I shows that a fire of this size emits about 82 KW of total radiation, Q_r.

Thus the observation of this fire by color photography would base the determination of size and shape of the radiating region upon only about 0.23 percent of the total radiant emission. It is possible that, for this fuel system, the boundaries of the radiator might still be defined with adequate accuracy by this technique; however, further investigation certainly is warranted to assure this. And it is entirely possible, for particular types of possible new flame agents, that this will not afford a valid description of the radiating source.

2.2 Indoor Still-air Heat Balances

Some preliminary heat balances were run on still-air fires of two fuels -- NTS and hexane -- in two diameters -- 18 and 22½ in.-- to ascertain what degree of agreement with input energy would be obtained. Simplifying assumptions made were (1) spherical symmetry of radiation intensity; (2) complete combustion of the fuel to CO_2 and H_2O vapor; (3) no loss of convective energy, Q_c, to the duct between the fire and the point of temperature measurements; (4) energy input to the exhaust stream from the blower was negligible; and (5) reflection of radiation from the soot-coated fiberglass cloth shrouds was negligible.

The system used (see Figure 1) was particularly convenient for determination of Q_c because it afforded a well-mixed exhaust stream which permitted measurement only of one temperature that was a reasonably true bulk temperature. All the products of combustion, plus any infused diluent air, passed through the exhaust blower, where good mixing of different-temperature portions of gas occurred. Temperature of the exhaust gas was measured downstream of the blower. Heat losses from the air in the short duct leading to the blower should be small because of the inherently low film coefficient of air, especially around the outside of the uninsulated duct where only natural convection existed. Temperature drop from this loss was estimated to be less than 1°F. The same natural convection film outside the blower housing would restrict heat loss there. And work input from the blower (3 HP motor, but throttled flow would reduce the amount of work done by the blower) was estimated to be very nearly equivalent to the heat losses to the walls. In any case, since Q_c for all fires fell between 150 and 250 KW, each of these nearly self-compensating effects constituted only about 1-2 percent of Q_c, and the net difference could safely be neglected.

The results of these tests are summarized in Table I.

TABLE I

PRELIMINARY HEAT BALANCES
(Indoor Fires)

Fuel	Power of Combustion $\bar{P}_c$, KW	Radiated Power (at 5 ft) $\bar{Q}_R$, KW	Convected Power $\bar{Q}_c$, KW	$\bar{Q}_R/\bar{P}_c$ %	$\bar{Q}_c/\bar{P}_c$ %	SUM $\frac{\bar{Q}_R + \bar{Q}_c}{\bar{P}_c}$ %
18-IN. PAN						
Hexane	267	66.5	200.5	25	75	100
NTS	234	81.6	155	35	66	101
22½-IN. PAN						
Hexane	322	86.8	251	27	78	'05
NTS	354	101.7	251	29	71	100

NOTE: All values are averaged over the entire run.

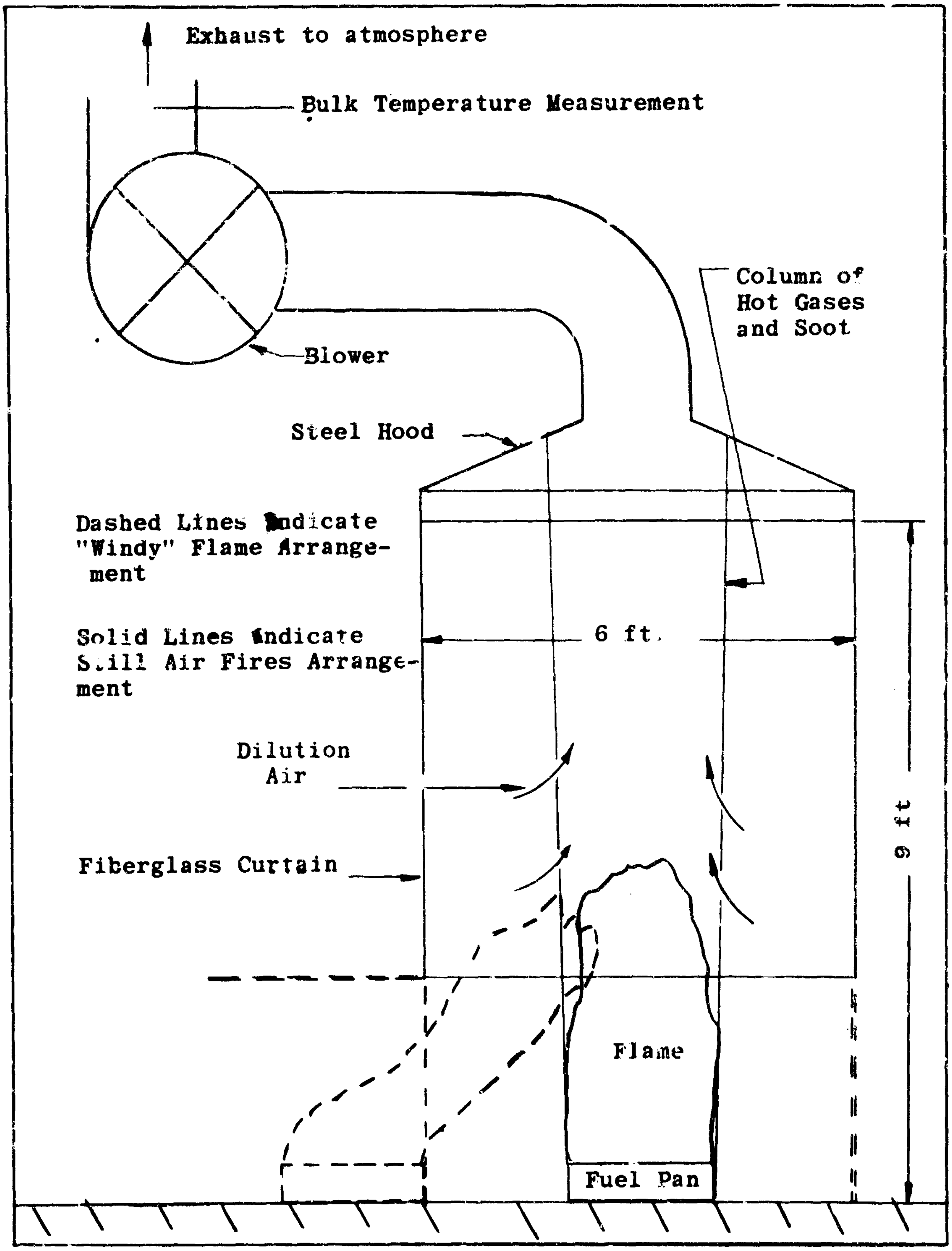

Figure 1. Indoor Heat Balance (and "Windy Flame") System

The extraordinarily good agreement, both within any one test and between tests, of output with input is probably due in large degree to the well-mixed, homogeneous exhaust stream. Values of $(\bar{Q}_r + \bar{Q}_c)/\bar{P}_c$ of 90-110 per cent would have been considered good, especially for such unrefined data. It is anticipated that considerably larger discrepancies will be encountered when larger fires are conducted outdoors and Q_c must be measured by sampling an unmixed, inhomogeneous thermal column.

2.3 "Windy Flame" Heat Balances and Gelled Fuel Tests

The system shown in Figure 1 also permits the simulation of controlled low-velocity winds by extending to the floor the fiberglass curtains on three sides of the hood, and carrying the fourth ("windward") curtain out horizontally above the pan. The curtains then cause air to enter the shrouded region principally via the opening in the windward side, and thus generate a smooth unidirectional air flow, or wind. The fuel pan is situated under the horizontal portion of the curtain, away from its normal central position under the hood. These arrangements are shown in dashed lines in Figure 1.

Measurements of Q_r were taken at 90° increments relative to windward. However, provision had not been made for mounting the sensors properly to observe the windward and leeward sides of the flame, and these measurements will have to be repeated. For the readings of Q_r normal to wind direction, it was surprising to find (at a wind velocity of ca. 2 ft/sec) that Q_r fell within the experimental variation of Q_r for still-air fires although the flame was tilted over to angles of about 45° to the vertical. Similarly, Q_c exhibited no significant change. A more thorough investigation of this lightwind effect is planned.

When gelled fuel became available, some preliminary measurements of still-air fires with this fuel (gelled NTS) were made on indoor fires of 18-inch diameter. The burning rate of NTS containing 3.65% by weight of M1 thickener was approximately 2/3 that of the liquid fuel (2.5 mm/min. vs. 4 mm/min.), and both Q_c and Q_r were lower by approximately equivalent amounts. There appeared to be no significant difference between the spectra of gelled and liquid NTS and no significant change in the ratio of Q_r/Q_c.

2.4 Gelled Fuel Preparation

The shipment of M1 thickener received early in this quarter permitted the preparation of thickened fuels for use on this program. Two pilot batches (nominally 22 gal each) of gelled fuel were made with ordinary gasoline to check out the procedure and to familiarize the operators with the process. The composition of

the first of these batches was approximately that specified for the portable flamethrower, 4% M1 (actual content was 3.65%, since the can of thickener did not specify the weight contained); the second was 2.0% M1. It was found that very little thickening of the fuel occurred within the first 7 minutes of agitation (air-powered Lightnin' propeller mixer), during which the thickener was added slowly and continuously. After an additional 10 minutes, the mix looked somewhat like lumpy chocolate syrup (in consistency, not color). After a total of 35 minutes, the fuel appeared quite well gelled, although not homogeneous. Propeller agitation was stopped, the drum was sealed and placed on the Morse rotator, and rolled for about 24 hours. The product gel was very clear and homogeneous, quite sticky, ropy, and elastic. This preparation procedure (in a heated area dehumidified to less than 20% R.H., with fuel at ambient temperature) was standardized arbitrarily for all succeeding mixes.

A 22-gal batch (nominal volume; actually prepared by weight as 138 lb of fuel) of NTS gel at 3.65% M1 was then prepared according to the standardized procedure. This gel appeared similar to the first gasoline gel, although no quantitative measurements of consistency were made. The gel was transferred (with some difficulty) into 5-gal glass carboys for dissemination tests and, in the case of the partial fifth carboy, for shipment to the indoor flame test site.

It should be noted that both gasoline and NTS gels were examined after 3 weeks storage at 60-80°F, and very significant losses in viscosity and adhesiveness were observed (again, no quantitative measurements were made). None of the gels had been peptized, since they had been prepared above the temperature at which this was recommended. Therefore, it would seem advisable, in order to obtain as high a degree of reproducibility as possible, to use freshly-prepared gels for all standard tests.

2.5 Explosive Dissemination Technique Tests

An initial series of dissemination tests was conducted during this period. These tests were run with standard 5-gal glass carboys filled from one 22-gal batch of 3.65% M1-thickened NTS gel (approximately the concentration specified -- for gasoline -- for use in portable flamethrowers). The carboys were suspended approximately 5 ft above the ground at the end of a "well sweep" consisting of a 20-ft length of 2-in pipe supported at its center and counterbalanced. In the first test, four Engineer's Special (J2) blasting caps were positioned in the approximate center of the carboy, with three caps oriented radially, horizontal, and one pointing straight down. Approximately 5 g of black powder (A5, fine) in a polyethylene bag, ignited by an M2 squib wired in parallel with the caps,

was hung beside the bottle as an igniter. When fired, the bottle was shattered into pieces no larger than a silver dollar. The fuel was dispersed in large discrete "blobs" in a random, non-symmetric pattern over an area about 16 yards in diameter; most, but not all, of the gel was ignited. The dispersion was considered excessive and undesirable, and the gel appeared to be excessively thickened.

The second test used one J2 blasting cap at the center of the carboy, pointing downward, and a similar igniter except that A1 black powder (coarse granules) was substituted to obtain longer burning. Bottle fragmentation was very similar to the prior test except that the base (which is almost 1/2 inch thick) remained intact. Dispersion of the gel appeared to be in a nearly continuous, umbrella-shaped (convex upward) sheet which covered a ground area about 4 ft x 6 ft. It is possible that the blasting cap was not properly centered, or that bottle wall thickness nonuniformities caused the slight asymmetry. Ignition was complete, and the main fire lasted for about four minutes. Except for the moderate asymmetry, the dispersion looked very good for our purposes.

A third test was made similar to the second except that the cap was located outside the bottle, centered on the base and pointing upward against the glass. The intent was to obtain a symmetrical deposit without having to open the carboy and insert any object. This would be valuable in the case of an experimental fuel which had been shipped by a contractor for evaluation, and which was either air-reactive, highly corrosive or toxic, etc. Unfortunately, the single cap was unable to defeat the massive glass bottom, although the side wall was ruptured at several points at the junction with the base and the gel slowly drained out after the igniter had burned out. This test was considered merely inconclusive and indicative that further investigation should be conducted.

2.6 Large Fires

The "windproof enclosure" at the outdoor Flame Range was fitted with a horizontal annular convection baffle to minimize wind and thermally-induced downdrafts and the base of the 4-mil polyethylene wind barrier was raised about 1½ feet off the ground to match air entrance area with exit area through the convection baffle. The enclosure was found to exhibit moderating effects upon ambient breezes, but winds of a few miles per hour caused lateral deflections of the flame of half a diameter or more. Radiation intensity at the periphery of the enclosure was sufficient, even from 2-ft diameter fires, to require use of a field stop on the Eppley thermopile to keep energy input within the permissible operating range (ca. 250 mw/cm^2 continuous). Weather conditions were not favorable after completion of the convection baffle, and no quantitative data were obtained from 4-ft diameter fires during this period.

2.7 Narrow-field-of-view Optics

A system of foreoptics which would afford a narrow field of view was desired for use with both the Eppley thermopile and the Perkin-Elmer spectrometer. This would permit the selective observation of small discrete zones in either the flame or the thermal column, either for total radiation or for spectral distribution.

A 4-inch diameter, f/1 Cassegrain spherical mirror system (Barnes R4D1), other than that associated with the Barnes radiometer, was available, but without any suspension or focusing mounts. A mounting was designed which would provide the desired fixed paraxial focus (focused to a point at infinity); thus the field of view will be annular, 4.00 in. O.D. x 2.125 in. I.D. This will permit observation of reasonably small regions, yet afford some averaging tendency to smooth out very localized fluctuations. A lens of arsenic trisulfide glass ("Servofrax", Servo Corp. of America), which transmits from 0.59 μ to about 12 μ, will be used to focus the concentrated radiation into either a parallel beam for the spectrometer or into a spot of proper size and position to impinge totally on the sensor disc of the Eppley.

Design of this mount has been completed and construction presently is being pressed. It is anticipated that this system will provide information of considerable interest concerning the amount and spectral nature of radiation from discrete regions of the fire.

3. Discussion of Findings and Conclusions

In the work to date, certain recurrent problems and conclusions have developed. Two of these are discussed separately in the following sections.

3.1 Fluctuations of Still-air Flames

Our work to date has been plagued by one major recurring problem -- the constant fluctuation of nominally steady flames. This can be seen in Figure 2, which shows an oscillograph record of a 60-second interval during the steady period of an 11-inch diameter NTS fire (similar records have been obtained in all fire sizes up to at least 4 ft diameter). This is a measurement of the broadband radiation at a point five feet from the fire, and clearly is anything but steady. The higher-frequency ripple varies between roughly 1 and 5 cps, with occasional rates of 10 cps or higher (seen on other records with expanded time scales), while the lower-frequency larger-amplitude fluctuations vary in period between 4 and 15 seconds.

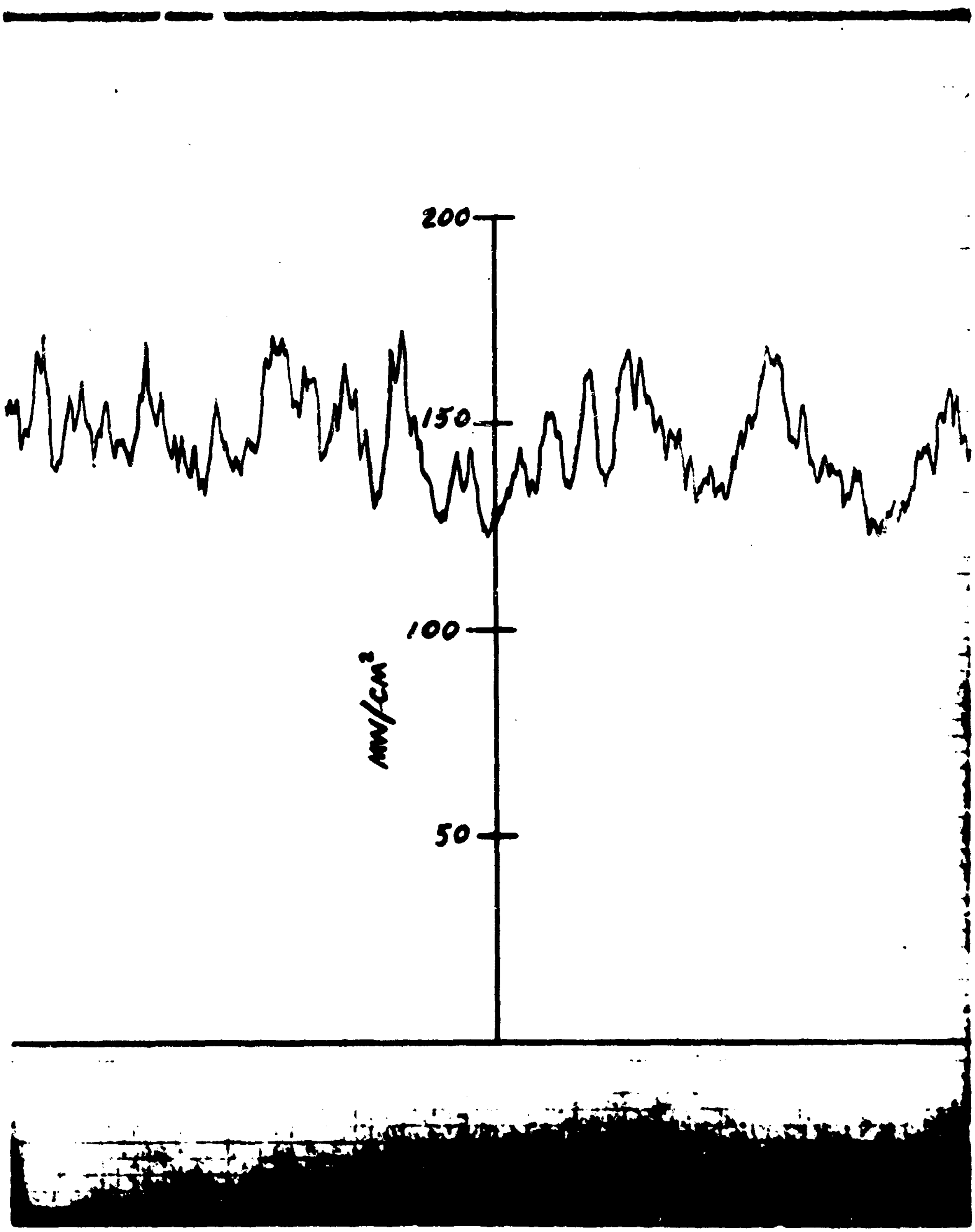

Figure 2. 60-Sec Oscillograph Record of "Steady-State" Radiation at 5 ft from 11-in. NTS Fire

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This kind of fluctuation has an effect not only on total radiant emission measurements, but also upon both spectral distribution of radiation (at least as measured by slow-scan spectrometer) and in convective energy data. In continuous monitoring of a parameter with fairly high-response sensors, as shown for total radiant emission, such oscillations about a mean value do not pose too severe a difficulty. But in a time-sampled system (one example would be a slow-scan spectrometer), these variations can introduce some uncertainties and inconsistencies by indicating a spurious peak or dip at say, a specific wavelength being observed at a given instant, which really was the result only of this random fluctuation about a steady mean value.

The effects of these oscillations upon convective energy measurements has yet to be determined, since our Q_C measurements to date all have involved fan-mixed exhaust gas streams. However, observations in the thermal columns of large outdoor fires necessarily will consist of readings taken at various radii at different instants of time; and it is reasonable to expect gas velocity and temperature at a point in the thermal column to vary with at least the major fluctuations in the fire. Hence it is to be expected that there will be inconsistencies in some of the thermal column profiles, i.e., that the effects of these oscillations upon the thermal column in free convection will be somewhat unfavorable.

The fluctuations which have been observed in nominally still air are severely aggravated, in the case of intended still-air fires outdoors, by the presence of even slight, almost unnoticeable breezes. Our test area very seldom experiences truly still air conditions for any significant period of time. We have attempted to provide some isolation from at least gentle breezes by means of an octagonal enclosure 20 feet across the flats by 25 feet high, with a transverse annular baffle near the top to reduce downward convection. The walls consist of 4-mil natural polyethylene film supported on both sides against wind loads by 4-inch-mesh wire screening; it has successfully withstood winds of over 35 mph. This barrier has produced some degree of suppression of gentle breeze effects, but is too small by almost a factor of two in height and perhaps by a factor of three or four in width.

There is no question in our minds that for quantitative comparisons of flame fuels, something equivalent to an empty airplane hangar would be immensely superior to an open field. Perhaps an economical substitute (which is beyond the funding limitations of this program) would consist of four to eight 50-foot antenna masts on an 80-100 ft minimum wall-wall distance, sheathed and roofed with screen-supported polyethylene which was adequately guyed to minimize flapping in wind gusts. This enclosure should have no larger an opening in the center of the roof than is required to remove combustion products; and ideally, for convective

:r)

energy studies, the exhaust port should be provided with a large-capacity, low-head fan to remove but especially to mix the combustion products to permit single-point, bulk temperature measurements.

3.2 Need for Evaluation by Rates, Not Totals

Apparently, the ultimate intent of this program was to develop a technique which could assess quantitatively the performance characteristics of a one-quart to five-gallon quantity of flame agent as functions of time during burning, and to relate instantaneous and total energy releases in all forms to chemical energy content of the agent. This would necessitate integration of each of the various quantities as a function of time. While such might be feasible for the radiation component, there are several parameters which do not lend themselves to this approach.

Unless Q_c is determined from the bulk temperature of a well-mixed exhaust stream, it is very unlikely that convective energy can be adequately monitored for an event whose mean output varies significantly as a function of time. A reasonable period of time is required to permit gas column velocity and temperature sensors to stabilize at the conditions existing at a given point in the column. Column parameters and diameters will change with a time-varying event; and column position is subject to lateral shifts even in still air from steady events. To monitor simultaneously the number of points across (with some necessarily outside of) the thermal column adequate to describe the instantaneous condition of the column would require a large number of sensors and recording channels, which would be quite expensive. In addition, the presence of this number of sensors in the thermal column almost surely would disturb the column flow excessively. The alternative technique, monitoring at one (or a few) point(s) and shifting the observation points with time, obviously is incompatible with a time-varying phenomenon.

Another difficulty is that of relating instantaneous rates of, say, Q_c and/or Q_r, to the mass rate of fuel consumption in a time-varying event. The mass burning rate surely will vary with overall fire size; or, Q_r and Q_c will vary with mass burning rate; but will the variation be in constant proportion? And how will this constancy, if postulated, be verified in a time-varying event? The situation is analogous to that of a solid-propellant rocket motor static firing in which one wishes to determine specific impulse. Generally the rocket motor cannot be weighed accurately and continuously during the firing. Hence all that is known about mass flow rate is the total weight of propellant consumed and the total time of consumption. If the burning surface was expected to be constant with time, the chamber pressure and thrust were constant, and the nozzle dimensions did not change, the mass flow rate can safely be assumed to be the

quotient of total weight divided by total time -- if the burning time was long with respect to starting and stopping transients. However, if any of the above conditions was not met to a fair degree, the calculated specific impulse will be only an average of a series of instantaneous values, and cannot accurately be related to performance in a substantially different system.

In contrast, during the firing of a liquid-propellant rocket the instantaneous flow rates of the propellants almost invariably are measured continuously. Thus, whatever transients may be encountered, if a steady-state condition is reached for only perhaps 0.5-2 seconds, the specific impulse under those particular conditions can be calculated accurately. The integrated totals may also be obtained if desired. But the performance under a set of conditions which existed only briefly, and which differed substantially from those which obtained during the major portion of the test, still may be accurately related to an entirely different system which would operate at the briefly-attained condition. Obviously, much more information, and of more reliable nature, may be obtained from measurements based upon rates than merely upon integrated total quantities.

For the reasons cited above, and for others directly paralleling them, it has become evident that quantitative assessment of flame agent performance would be greatly enhanced by measurement of parameters as rates of release at each of a series of constant, controlled agent flow rates and fire diameters. Thus the explosive dissemination technique which has been explored briefly and shown to be capable of providing a reasonably uniform (and presumably repeatable, although this has not yet been demonstrated) deposit, is not considered to be desirable from the standpoint of the requirements of this program.

It may be assumed, subject to verification experiments, that the "efficiency of combustion", however defined (e.g., percent of heat of combustion radiated as Q_r, ratio of Q_r/Q_c, percent of heat of combustion not liberated, etc.) of a given flame agent will vary only slightly as a function of fire size. If this is found to be true, then the transient phenomena related to the performance of a specific flame weapon can be monitored satisfactorily by measuring only the radiated energy, Q_r, and inferring both Q_c and the energy lost as incomplete combustion (Q_u) from Q_r via experimentally determined ratios of Q_c/Q_r and Q_u/Q_r for the specific agent. Since radiation is readily measured as a transient quantity, such an evaluation could be easily and economically made. Of course, this approach is valid only if the ratios of Q_c/Q_r and Q_u/Q_r are actually shown, for the specific agent in each case, to be relatively insensitive to fire size, time, etc.

4. Future Work

Future work can be divided into two general categories: indoor fires and outdoor fires. These are discussed separately below.

4.1 Indoor Fires

4.1.1 18-24 in. Diameter Pan Fires

Spatial distribution of radiation intensity over hemispherical control surface and base plane.

Mapping (with narrow-field-of-view foreoptics) of radiation intensity and/or spectral distribution along vertical axis and transverse thereto (including thermal column).

Sampling of combustion products for unburned species including soot (with simultaneous heat balances).

Determination of optical density of flame.

4.1.2 Burning Stationary Fuel Rod in High Velocity Air Stream

Burning Rate
Radiation characteristics

4.2 Outdoor Fires

4.2.1 Obtain spectra and radiation intensity measurements from still-air and/or windy flames of 4-ft diameter, plus burning rates.

4.2.2 All other items shown in 4.1.1.

4.2.3 Comparison of integrated total radiation from transient fires resulting from dissemination of liquid (very short duration) and gelled fuel, 5 gal quantities.